THE CLAIMS

What is claimed is:

1. A method of determining average grain size and grain size distribution of a polycrystalline material, comprising:

providing a sample comprising said polycrystalline material;

irradiating a measurement point on the sample with monochromatic radiation energy generated from a radiation source, wherein the beam size and divergence of said radiation energy is adjusted so that an adequate number of crystal grains of said sample is irradiated by the monochromatic radiation energy;

detecting radiation energy diffracted from the sample to capture a plurality of diffraction arcs within a single data capture frame;

generating average grain size and grain size distribution data from the diffraction data of the detected diffracted radiation energy, according to an integral grain size analysis protocol comprising the steps of:

(a) digitally registering raw diffraction data from the plurality of diffraction arcs captured;

(b) filtering the registered diffraction data to remove background noise, exclude diffraction overlaps or truncations, and compensate for biased data obtained from regions with highly preferred orientations; and

(c) correlating average grain size and grain size distribution data with the filtered diffraction data.

2. The method of claim 1, wherein the monochromatic radiation energy generated by the radiation source is monochromatic x-radiation.

3. The method of claim 1, wherein the step of detecting radiation energy diffracted from the sample is conducted in proximity to the sample measuring point at a detection locus in fixed spatial relationship to the radiation source.

4. The method of claim 1, wherein the sample is moved only in the sample plane during data acquisition.

5. The method of claim 1, wherein the sample comprises a textured polycrystalline material having grain size in a range of from about 0.1 micron to about 100 microns.

6. The method of claim 1, wherein the step of digitally registering the raw diffraction data comprises:

acquiring and storing the raw diffraction data in the form of a spot intensity distribution image;
converting the spot intensity distribution image from χ and 2θ coordinates to x and y coordinates;
outputting the converted spot intensity distribution image in a three-dimensional surface plot form, with diffraction intensity of each spot registered as spike height at each x and y location.

7. The method of claim 1, wherein the step of filtering the registered diffraction data comprises using minimum intensity criteria to remove background noise.

8. The method of claim 7, wherein a three-dimensional peak-searching algorithm is employed to search for spots meeting the minimum intensity criteria.

9. The method of claim 1, wherein the step of filtering the registered diffraction data comprises fitting said registered diffraction data against a standard distribution function to exclude spots with either excessive intensity overlaps or intensity truncations.

10. The method of claim 9, wherein the standard distribution function comprises a two-dimensional Gaussian curve.

11. The method of claim 1, wherein the step of filtering the registered diffraction data comprises excluding biased data obtained from regions with highly preferred orientations.

12. The method of claim 1, wherein the step of correlating average grain size and grain size distribution data with the filtered diffraction data comprises:

providing a set of standard polycrystalline samples with known average grain sizes and grain size distributions; and

calibrating the filtered diffraction data of said sample comprising said polycrystalline material, against the diffraction data of said standard polycrystalline samples, to generate corresponding average grain size and grain size distribution for said sample comprising said polycrystalline material.

13. The method of claim 1, wherein the step of correlating average grain size and grain size distribution data with the filtered diffraction data comprises:

determining a total number of grains within an irradiated volume of the sample;

calculating average grain size from the total number of grains within the irradiated volume;

converting an intensity distribution for the sample into a grain size distribution, based on the average grain size calculated.

14. The method of claim 13, wherein the total number of grains within the irradiation volume of the sample is determined upon mathematically factoring in presence of texture in the sample.

15. The method of claim 13, wherein the intensity distribution is converted into grain size distribution by the steps of:

plotting the intensity distribution in the form of a lognormal graph showing grain count frequency versus natural logarithm of diffraction intensity of each grain;

setting the centroid of the plotted lognormal graph to be a natural logarithm of the average grain size calculated.

16. The method of claim 15, wherein the grain size distribution is further modified by plotting the grain count frequency versus natural logarithm of grain size of each grain divided by the average grain size calculated.

17. The method of claim 1, wherein the radiation source comprises a collimated x-ray source having means for adjusting beam size and divergence of the generated radiation energy.

18. The method of claim 17, wherein the collimated x-ray source comprises a sealed x-ray beam source, a monochromator, and a tapered capillary collimator.

19. The method of claim 1, wherein the radiation energy diffracted from the sample is detected by a two-dimensional area detector.

20. The method of claim 19, wherein the two-dimensional area detector is position sensitive.

21. The method of claim 19, wherein the two-dimensional area detector comprises means for transfer of detected diffraction data into electronic digital format.

22. The method of claim 19, wherein the two-dimensional area detector is selected from the group consisting of proportional counters, x-ray image charge-coupled device (CCD) cameras, and x-ray image plates.

23. The method of claim 19, wherein the two-dimensional area detector comprises a multiwire gas proportional counter.

24. A crystal grain size analyzing system for determination of average crystal grain size and size distribution of a polycrystalline material, said system comprising:

a sample comprising said polycrystalline material and defining an associated sample plane;

a collimated source of monochromatic radiation energy arranged to direct radiation energy to a measurement point on the sample, wherein said collimated source comprises means for adjusting beam size and divergence of the monochromatic radiation energy;

a 2-dimensional area detector that registers radiation energy diffracted from the sample at the measurement point, said collimated source of monochromatic radiation energy and said 2-dimensional area detector being in a fixed spatial relationship to one another and sufficiently

proximate to the sample measuring point to capture a plurality of diffraction arcs within a single data capture frame of said detector;

a sample motion assembly translating the sample in the sample plane; and

an integral grain size analysis processor constructed and arranged to generate average grain size and grain size distribution data from the detected diffraction data of the diffracted energy.

25. The crystal grain size analyzing system of claim 24, wherein the monochromatic radiation energy source emits monochromatic x-radiation.

26. The crystal grain size analyzing system of claim 24, wherein the sample motion assembly is constructed and arranged to permit only in-plane motions of the sample.

27. The crystal grain size analyzing system of claim 24, which does not include any Eulerian cradle providing χ rotation, or any θ-2θ goniometer component or apparatus.

28. The crystal grain size analyzing system of claim 24, wherein the sample comprises a textured polycrystalline material having grain size within a range of from about 0.1 micron to about 100 microns.

29. The crystal grain size analyzing system of claim 24, wherein the integral grain size analysis processor comprises computational means for:

(a) digitally registering raw diffraction data from the plurality of diffraction arcs captured;

(b) filtering the registered diffraction data to remove background noise, exclude diffraction overlaps or truncations, and compensate for biased data obtained from regions of highly preferred orientations; and

(c) correlating average grain size and grain size distribution data with the filtered diffraction data.

30. The crystal grain size analyzing system of claim 29, wherein the integral grain size analysis processor digitally registers raw diffraction data by the following steps:

acquiring and storing the raw diffraction data in the form of a spot intensity distribution image;

converting the spot intensity distribution image from χ and 2θ coordinates to x and y coordinates;

outputting the converted spot intensity distribution image in a three-dimensional surface plot form, with diffraction intensity of each spot registered as spike height at each x and y location.

31.

~~30.~~ The crystal grain size analyzing system of claim 29, wherein the integral grain size analysis processor filters the registered diffraction data by using minimum intensity criteria to remove background noise.

32.

~~31.~~ The crystal grain size analyzing system of claim 30, wherein the integral grain size analysis processor employs a three-dimensional peak-searching algorithm to search for spots meeting the minimum intensity criteria.

Rule 1.126

33

~~32.~~ The crystal grain size analyzing system of claim 29, wherein the integral grain size analysis processor filters the registered diffraction data by fitting said data against a standard distribution function to exclude spots with either excessive intensity overlaps or intensity truncations.

34.

~~33.~~ The crystal grain size analyzing system of claim ~~32,~~ 33 wherein the standard distribution function comprises a two-dimensional Gaussian curve.

35.

34. The crystal grain size analyzing system of claim 29, wherein the integral grain size analysis processor filters the registered diffraction data by excluding biased data obtained from regions with highly preferred orientations.

36.

35. The crystal grain size analyzing system of claim 29, wherein the integral grain size analysis processor correlates average grain size and grain size distribution data with the filtered diffraction data, by providing a set of standard polycrystalline samples with known average grain sizes and grain size distributions, and then calibrating the filtered diffraction data of said sample comprising the polycrystalline material, against the diffraction data of said standard polycrystalline samples, to generate corresponding average grain size and grain size distribution for said sample comprising said polycrystalline material.

37.

35. The crystal grain size analyzing system of claim 29, wherein the integral grain size analysis processor correlates average grain size and grain size distribution data with the filtered diffraction data, by the steps of:

2uA
1-12-6

determining a total number of grains within an irradiated volume of the sample;

calculating average grain size from the total number of grains within the irradiated volume;

converting an intensity distribution for the sample into a grain size distribution, based on the average grain size calculated.

38. 37

36. The crystal grain size analyzing system of claim 35, wherein the integral grain size analysis processor determines total number of grains within the irradiation volume of the sample upon mathematically factoring in presence of texture in the sample.

39. ~~37~~. The crystal grain size analyzing system of claim ~~35~~ 37, wherein the integral grain size analysis processor converts the intensity distribution into grain size distribution, by plotting the intensity distribution in the form of a log-normal graph showing grain count frequency versus natural logarithm of diffraction intensity of each grain, and setting the centroid of the plotted log-normal graph to be a natural logarithm of the average grain size calculated.

40. ~~38~~. The crystal grain size analyzing system of claim ~~35~~ 37, wherein the integral grain size analysis processor further modifies the grain size distribution by plotting the grain count frequency versus natural logarithm of grain size of each grain divided by the average grain size calculated.

41. ~~39~~. The crystal grain size analyzing system of claim 24, wherein the collimated source comprises a sealed x-ray beam source, a monochromator, and a tapered capillary collimator.

42. ~~40~~. The crystal grain size analyzing system of claim 24, wherein the two-dimensional area detector is position sensitive.

43. ~~41~~. The crystal grain size analyzing system of claim 24, wherein the two-dimensional area detector comprises means for transfer of detected diffraction data into electronic digital format.

ZLLA
1-126

44. ~~42~~. The crystal grain size analyzing system of claim 24, wherein the two-dimensional area detector is selected from the group consisting of proportional counters, x-ray image charge-coupled device (CCD) cameras, and x-ray image plates.

45. ~~43~~. The crystal grain size analyzing system of claim 24, wherein the two-dimensional area detector comprises a multiwire gas proportional counter.